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THIN FILM GAGES FOR IGNITION AND COMBUSTION EXPERIMENTS

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Thin film gages are of use in studies on the ignition and combustion of gas propellants. The work described in this report covers fabrication and calibration techniques for thin film gages on several substrates. These gages are intended to determine heat flux values in an experiment on the convective ignition of solid propellants.

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I. INTRODUCTION

Primarily, the intent of this work was to develop heat flux gages in special configurations for use in convective ignition studies of solid propellants. For the convective ignition work, it was desired to monitor the heat flux to various solid materials from various inert gases under "laminar" flow conditions over streamlined shapes. From these data, heat transfer could be predicted for solid propellant samples under similar conditions.

II. BACKGROUND

In his thesis work, Birk[1] studied the ignition of solid propellants when exposed to transient heating in a shock tube facility. This work considered the reaction of a cylindrical propellant sample with the gas flow perpendicular to the cylinder axis. Heat flux, gas flow, and gas composition were the observed parameters. The results of Birk's work led to the establishment of a task here to improve the modeling aspects of convective ignition and to extend the measurements to greater limits than were obtainable in the shock tube facility.

The work described herein covers fabrication and calibration techniques for thin film gages on several materials with configurations designed to meet modeling requirements of the convective ignition task.

It was desired to have heat flux gages with a range of thermal parameters so that heat transfer to solid propellant samples could be modeled under the flow conditions of this experiment.

The following basic conditions relevant to construction of the heat flux gage were established in discussions with N. Klein and A. Birk of this laboratory.

The propellant/gage samples are to be cylinders with one end finished as a radius matching hemisphere. The gas flow is to be coaxial with the cylinder axis and incident on the hemisphere.

The gas flow is to be laminar in the vicinity of the sample, and the sample is to be prepared as a streamline configuration.

Gas flow conditions are to be adjustable over a range of values. The following values are considered as the design goals:

Temperatures to 1000 C,
Pressure up to 2MPa (20 ATM),
Flow velocities to 150 m/s,
Long exposure times (e.g., 200 ms at 150 m/s).

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[1] *A. Birk and L.H. Caveny, "Convective Ignition of Propellant Cylinders in a Developing Cross-Flow Field," Princeton University, September 1980, MAE Report No. 1486.*

Discussions with A. Birk indicated that the heat flux gages to be used at the extreme conditions should be constructed to withstand and monitor surface temperatures up to 400 C. This places strong emphasis upon the method of attaching the leads to the gage body.

The sample dimensions had been previously selected as a cylinder diameter of 5.56 mm, with a matching hemispherical end. The sample length was set at 40 mm.

Surface finish was not specified but was expected to be a "polish" with no major discontinuities upon which turbulence could be established.

III. MATERIAL SELECTION

A. Substrate Material

Selection of substrate materials had been tentatively made by N. Klein as Macor and boron nitride.

Macor is a machinable ceramic made by Corning. It may be machined with standard drill and lathe tools.[2,3,4]

An option of using Pyrex, a borosilicate glass, was held open because this material has been well characterized in previous use as a heat flux gage substrate.[5] Its only drawback was the more complex fabrication techniques required to obtain specific shapes and sizes. In reality, the cylindrical shape with a hemispherical end is easy to obtain. Pyrex rod of 5.4 mm diameter was obtained. By careful melting of one end, a hemisphere was established by surface tension.

A number of plastics were selected for use in the heat flux tests, but not as substrates for the heat flux gages. It was decided to search for high temperature plastic materials that could survive for at least a portion of a test and could provide a set of thermal properties closer to those of a propellant than would be obtained with a ceramic substrate.

Samples of Vespel were obtained for test purposes. Vespel is a high temperature plastic from Dupont. It has a decomposition temperature of 400 C.

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[2] *"Macor Machinable Glass Ceramic," Corning Glass Works, Corning, NY.*

[3] *D.G. Grossman, "Machining a Machinable Glass-Ceramic," American Machinist, May 1978.*

[4] *"Macor Machinable Glass Ceramic," Accuratus Ceramic Corp., Washington, NJ, Bulletin 803.*

[5] *G.T. Skinner, "Calibration of Thin Film Gage Backing Materials," ARS Journal, p. 671, May 1961.*

Zircon and Vycor have been used as heat flux gage substrates.[6] Some difficulties were encountered in stabilizing the gages on Zircon.

Sintered alumina is a candidate material. It is used as a substrate material in commercially available platinum resistance thermometers.[7] The hardness of the material makes the fabrication of a hemispherical end somewhat difficult.

B. Film and Lead Materials

Platinum has been the traditional choice for heat flux gages for a number of good reasons. The platinum film is easy to obtain on glass by the pyrolytic technique with commercial preparations (e.g., Hanovia Liquid Platinum). The metal film of platinum is a noble metal so it is relatively immune to damage from flames or the environment (oxidation).

Water and water vapor will affect thin films of platinum. The bonding of the platinum to its substrate is affected, and the resistance may change. For inert gas atmospheres, as are planned in the convective ignition experiment, reactivity of the metal film is only significant for the short time of the assembly of the test apparatus. During this time, the gage will be exposed to air at room temperature.

The requirement of reliably sensing surface temperatures of 400 C is probably restrictive of many metals as this is a significant fraction of the melting point of bulk metals, and recrystallization of the film may start to be a problem.

Gold is a noble metal that is easily sputtered in an adherent film and may be a good gage material if tests indicate that the calibrations at higher temperatures are stable.

In the final analysis, platinum was arbitrarily chosen based on its previous use with pyrex heat flux gages and the easy availability of the alternate techniques of application by sputtering or by pyrolysis of compounds.

Calculations were made for a wide range of candidate film materials to establish the resistance range that could by achieved for various thicknesses of film. In Table 1, the resistances to be expected at two different thicknesses are listed. These calculations are based on the resistivity of bulk materials. For films of 100 nm thickness, the value of the resistivity should be expected to increase.

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[6] *J.A. Keller and M.W. Ryan, "Measurement of Heat Flux from Initiators for Solid Propellants," ARS Journal, p. 1375, Oct 1961.*

[7] *"Temperature Measurement Handbook," Omega Engineering, Inc., Stamford, CT, 1981.*

Table 1. Gage and Lead Materials

	T C	ρ μΩ-cm	R_1 Ω	R_2 Ω	TCR	MP C
Al	0	2.63	1.3	6.5	0.004	659
C	0	5000	2500	12500	-.000	(3500)
Cr	28	13.0	6.5	32.5	0.003	1890
Cu	20	1.69	.85	4.25	.0068	1083
Au	20	2.44	1.2	6	0.004	1063
C (Graphite)	0	800	400	2000	-----	----
Mo	20	5.7	2.85	14.3	.004	2500
Ni	20	6.84	3.42	17.1	.0069	1452
Pt	20	10	5	25	.004	1755
	100	14.8	7.42	37.1	-----	----
	400	26	13	65	-----	----
Si	20	58	29	145	-----	1420
Ag	0	1.5	.75	3.75	.004	960
	100	2.15	1.07	5.35	-----	----
Ta	27	13.85	6.92	34.6	.0038	2850
W	20	5.51	2.75	13.8	.004	3400
	727	25.3	12.6	63	-----	----

$R = \rho \times \ell / (t + w)$
$R = R_o (1 + TCR \times T) = R_o (1 + \alpha T)$
R_1: ℓ (1 cm), t (200 nm), w (1 mm), length (ℓ), thickness (t)
R_2: ℓ (1 cm), t (40 nm), w (1 mm), width (w)
ρ: Resistivity
TCR: Temperature coefficient of resistance
MP: Melting point

As leads from the sensor area, a metal film, along the sides of the cylindrical section, provides the least interference with the flow pattern. In the hottest and highest stress areas, film leads suffer less damage than alternate methods of lead attachment to the sensor area.

The primary requirements for the film lead material are moderately good electrical conductivity, stability upon thermal cycling and good bond strength to the substrate. Platinum and gold are the only metals seriously considered in this work.

Platinum was generally selected for the four wire lead configuration where lead resistance was not critical to the gage functioning. Platinum was applied as leads by the same techniques used to deposit the sensor area.

Gold was tested because of its higher electrical conductivity. Caution must be exercised in applying gold near the sensor area as the gold will alloy with the platinum during sensor annealing.

IV. THIN FILM DEPOSITION TECHNIQUES

A. Alternatives

Thin metallic films may be deposited on ceramic substrates by a variety of techniques. These include chemical deposition, evaporation, and cathodic sputtering. The technology is reviewed by Chopra.[8]

It was decided to utilize sputtering techniques based on the range of materials that could be handled, the potential ease of using masks to define the deposition area, and the probable good control of the film thickness.

The pyrolysis of platinum compounds (e.g., Hanovia Liquid Platinum) is a standard, well established technique[9] for making thin platinum resistors on glass substrates. The technique has been used extensively for the fabrication of platinum heat flux gages on Pyrex.[1] The major drawback with the pyrolysis technique is a difficulty in obtaining thicker, low resistance films for leads and the associated repetitive firing of the gage body to temperatures near the softening point of Pyrex.

V. SPUTTERING APPARATUS AND TECHNIQUES

A wide range of options is available in sputtering apparatus. The technology is reviewed by Chopra.[8] In its simplest form, cathodic sputtering consists of establishing a glow discharge between two electrodes in a moderate vacuum. The positive gas ions impact on the cathode and drive cathode atoms into the glow space. A substrate in the vicinity of the cathode will collect a portion of the sputtered atoms, and a film will be formed.

The sputtering technique offers one of the best methods of obtaining an atomically clean surface. If the substrate material is conductive, it is used as the cathode in a sputtering device, and the surface layer of atoms is sputtered away, including contaminant and bound materials. If the material is not a conductor, a charge will build up on the surface, and the positive ions will be deflected. Various techniques have been developed to modify the sputtering apparatus to neutralize the surface charge at regular intervals or to screen the blocking charge.

With the simple diode type sputtering unit, a mask (e.g., screen) must be used over the ceramic material. The conducting mask is placed in contact with the nonconducting substrate and is electrically connected to the cathode. The positive ions of the glow discharge are accelerated towards the mask and a fraction pass through the openings to impact on the substrate. With this technique, the mask must be moved to clean an area. The substrate under the closed portions of mask is not impacted by the ions.

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[8] *K.L. Chopra, "Thin Film Phenomena," McGraw-Hill Book Company, New York, 1969.*

[9] *J. Strong, "Procedures in Experimental Physics," Prentice-Hall, Inc., Englewood Cliffs, NJ, 1938.*

Normally, an inert gas, such as argon, is bled into the system at a low rate as the vacuum pump is kept running. This provides a specific gas for the glow discharge and tends to prevent back streaming of oil from the vacuum pump. However, even high purity gases supplied commercially contain significant levels of water and oil vapors. A cold trap is normally used to remove these contaminants, and in "good" systems a getter trap is used to remove reactive atoms (oxygen).

A commercially available diode type of sputtering unit was available within the laboratory (K. White), and this was available for use on the thin film gage fabrication tests.

The unit was a Balzers Union "Sputtering Device." Normally, this apparatus is used to sputter conductive films onto electron microscope samples. As it was obtained by us, it had no cold trap or getter section. Most of the early work was done without utilizing a cold trap despite the clear warnings in the literature.[8] The system readily deposited "nice" films of gold and platinum on glass slides. An extended learning period was spent in observing and testing platinum and gold films on glass slides, Pyrex tubes, and Macor samples. Surface polishing, cleaning, and masking techniques were studied for the various substrate materials and shapes.

The basic sequence of operations used in depositing a metal film by sputtering will now be described.

The sputtering apparatus has been precleaned to minimize contamination of the substrate. A sample of the metal to be sputtered onto the substrate is placed in its electrode retainer position. A substrate specimen is polished, cleaned chemically and placed on the stage of the sputtering apparatus. The substrate is covered with a screen so that it touches the supporting electrode. This screen is used during the etch cleaning cycle.

The system is assembled and then evacuated. When the pressure has dropped to 10 Pa, argon is flushed through the system to remove volatile contaminants and residual air. The argon pressure is allowed to rise nearly to atmospheric pressure. The system is again pumped down to 10 Pa, and the flow of argon is adjusted to maintain this pressure. A constant sweep of argon tends to remove contaminants that are released during the glow discharge cycles.

At this point, a cold trap should be activated in the inlet tubing for the argon feed.

The electrode upon which the substrate rests is now polarized so that the positive argon ions will strike the screen and substrate sample.

A time is set to control the duration of the etch cycle, and the cycle is initiated. The voltage is adjusted to give the desired discharge current (35 mA max). The etch cycle is repeated several times with movement of the substrate and screen between runs. After the etch cycle is completed, the screen is moved out of the way or removed.

Several options are available at this time. The substrate may have one of the masking techniques applied, or metal may be sputtered over the entire

substrate area. With the latter approach, contamination of the sample by handling or masking materials is prevented.

Using no mask, the sensor area is placed in a position for optimum film deposition. The system is flushed, and the pressure of argon is adjusted to 5 Pa. The metal sample to be sputtered is sputter etched to remove any surface contamination. A shutter is used to prevent contamination of the substrate area during the target cleaning operation.

The time and discharge current are selected for the sensor sputtering cycle. For platinum on Pyrex sensors of 40 ohm final resistance, a 2-minute cycle at 35 mA was used. After the sensor area is sputtered, a mask may be placed over the sensor area to prevent further metal deposition in this area. More metal may be sputtered to lower the resistance of the sensor leads.

After the sputtering operation, the film may be removed from specific areas to define the sensor area and gage lead areas. With the configuration used in this work, the film was removed by polishing with a fine alumina powder applied with a small "stick" of phenolic or dense wood. The polishing operation was performed in a dry state to prevent moisture interacting with the metal film.

After the gage and lead areas are defined, the gage must be fired/annealed to stabilize the film resistance and film adhesion. The annealing process allows the crystallinity of the metal in the film to develop. During the sputtering process, defects and absorbed materials are frozen into the film. Chopra[8] states that the preferred annealing temperature is close to the recrystallization temperature of the metal in the film. It must be remembered that the properties of very small particles (crystals, or amorphous layers) may differ appreciably from those obtained in bulk samples. It appears that the proper annealing temperature and time cycle are generally obtained by experiment.

The annealing processes are bounded by a limiting temperature at which agglomeration of the film occurs. In this case the metal of the film condenses into individual particles that are electrically separated from each other. The resistance of the film increases dramatically and may approach the resistance of the substrate before a film was deposited. With platinum films on Pyrex, allowing the specimen to remain near the softening point of Pyrex (800 C) caused a rapid increase in resistance. The film would still appear visually good, but with extended heating the resistance jumped and the film became visually diffuse. It appeared as if the film had diffused into the glass surface.

After the annealing process, the lead wires are attached, and the temperature calibration cycles are run.

VI. PLATINUM FILM BY PYROLYSIS TECHNIQUE

The development of thin platinum films on glass or ceramic substrates by pyrolysis of platinum compounds is a well established technique.[9] The technique described by Birk[1] was used to compare the pyrolysis technique with the sputtering technique.

Basically, this method consists of the following steps:

Chemically clean the substrate.

Define the area to have a metal film by masking with a plastic tape, airbrush with Hanovia Liquid Platinum diluted with chloroform (25% Hanovia Liquid Platinum).

Air dry for 30 minutes.

Oven anneal to a temperature of 725 C.

The equipment required to make the gages with the Hanovia liquid is simple and inexpensive relative to the sputtering method: polyester tape for masking, an airbrush to spray on the thin layers of Hanovia liquid, a furnace capable of reaching temperatures of about 800 C and a temperature sensor to monitor the annealing temperature.

On Pyrex, at least, the resistance of the sensor element may be modified by repeated application of the liquid platinum and annealing to decrease the sensor resistance. To increase the resistance of the film, cautious annealing of the structure to higher temperatures causes partial agglomeration. My experience indicates that it is preferable to avoid the process of increasing the film resistance by agglomeration. An accurately controlled oven (time and temperature) might make the process more reproducible.

A Pyrex tube with one end fused into a hemisphere was used for the first tests. A tape mask was applied to define a sensor 1 mm wide and 3 mm long. One part Hanovia liquid and three parts chloroform were sprayed by airbrush from a distance of 20 cm. Four layers were sprayed on and allowed to air dry. The mask was stripped away, and a thermocouple was inserted into the tube to monitor the sample temperature during the annealing cycle. The tube was inserted slowly into a preheated tube furnace. The Hanovia film first chars, and then the organic binder burns away, leaving a silvery film. The thermocouple temperature was allowed to rise to 725 C and held for one minute. The sample was cooled slowly at the mouth of the furnace. The annealed sensor resistance was in the neighborhood of 100 ohms. Twenty percent variations were obtained in sensor resistance by unplanned variations in application technique. By practice, this variation could probably be reduced to several percent.

The chemical cleaning procedure for glasses normally involves an acid bath to leach alkali ions and metallic contaminants from the surface. A bath of hydrochloric acid was used for most of the Pyrex samples. In tests with the Macor substrate samples, acid baths produced surface pitting as is pointed out in the technical pamphlets. Balzers substrate cleaning solutions were used for cleaning the Macor samples.

Macor samples coated with the Hanovia Liquid Platinum produced sensor elements with a lower temperature coefficient of resistance than the Pyrex gages. It was also necessary to anneal the Macor to higher temperatures (800 C to 900 C) in order to stabilize the resistance.

VII. GAGE LEAD CONFIGURATION

The standard two wire gage is subject to numerous problems that influence the accuracy of resistance measurements.

If the leads from the sensor element are exposed to temperature variations, the temperature coefficient of resistance of the leg resistance will modify the signal from the sensor element.

Normally, during calibration, the entire gage is allowed to stabilize in a "bath." This causes the resistance of the leads to stabilize at the same temperature as the sensor, and the resulting calibration plot is valid for long term temperature variations. If the gage is now inserted into an experimental fixture where very rapid changes are to be followed, the leads will not change at the same rate as the sensor, and this causes an error in the detected signal.

The lead errors may be minimized by making certain that the resistance of the sensor is much greater than the resistance of the leads. In Birk's work,[1] holes were drilled in the Pyrex tubing immediately adjacent to the sensor area, and "large" wires were connected to the sensor through these holes. The gage was then calibrated as a unit in a furnace. No estimate is given for errors due to lead heating.

The only effective way to avoid the lead error problem with the two wire gage is to make the sensor element with a high resistance (500 Ω).

With the Macor samples in which a sensor was to be placed on the stagnation zone of the hemispherical end, a hole would be required through most of the length of the sample. This caused the bodies to be fairly sensitive to breakage in use. An additional problem area is the stability of the material used to fill the holes, when exposed to the long-term hot gas flow to be expected in the convective ignition tests. Erosion of the material in the holes could (probable) cause a disturbance in the flow pattern that would initiate turbulence.

An alternate lead configuration eliminates most of the problems to be encountered with a two wire gage. The four lead gage or four wire gage configuration consists of two leads to each end of the sensor element. In a circuit one of the leads on each end acts as a current carrying lead. The other lead on each end serves in a voltage sensing circuit. The voltage sensing leads are used to detect the voltage difference across the ends of the sensor due to the current in the current leads. In use, both the current and voltage are monitored so that the resistance of the sensor can be determined by a simple division.

The derived resistance of the sensor in the four wire configuration is essentially independent of the resistance in the current or voltage sensing legs.

Using the four wire gage configuration, thin film leads may be run from the sensor element down the outside of the sample. Grooves or holes may be cut into the bottom end of the streamlined body to lead the thin film into a covered area for wire attachment. In this manner, the surface of the body has no protrusions or cavities for turbulence generation.

The primary advantage in using the four wire gage is that calibration to accuracy better than 1% is feasible and that the calibration is related to the sensor element only. Heating of the gage legs does not influence the gage in calibration or in use. A steady state calibration of sensor resistance should be the same as would be achieved with an ideal dynamic calibration.

VIII. LEAD WIRE ATTACHMENT

Lead wire attachment to the gage body presents a number of problems that are unique to this convective ignition task. Turbulence generating protrusions are not allowed on the surface of the gage that is exposed to the hot gas flow. The surface of the gage is expected to attain temperatures of 400 C and measure that temperature accurately (<1% desired). It is to be expected that vibration of the gage body will occur at the higher flow rates. It is desired that the gage survive more than one test and preferably more than twenty tests with no more than recalibration being required.

As a general rule, the lead should be trapped by the material of the substrate to prevent stress on the electrical joint. This wire joint stress may be due to mechanical vibrations or due to differing coefficients of thermal expansion.

Bonding a lead to the exterior surface of the cylinder produces a joint that will be subjected to the stresses of the lead motions. Either the joint will fail easily (silver paste), or the film adhesion to the substrate may fail (solders). In large thermal stresses to be encountered, the joint is also subjected to expansion of the metal and failure of the bond by this cause.

With either the sputtering technique or the pyrolytic film, holes or grooves in the substrate may be metalized so that the film is led away from the exposed surface to form the bond with the lead.

Birk[1] cemented wires into platinum coated holes with a silver paste. The holes provided an anchor point for the wires to minimize the strain on the thin film/silver paste joint. He reports excellent results and good durability were obtained in the shock tube tests. The temperature limits of the conducting silver pastes is in the range of 170 C to 200 C, depending on the formulation of the binder. Calibration of the gage must therefore be limited to 200 C to prevent decomposition of the silver paste. Under the dynamic conditions of a test, the surface temperature of the sensor can be appreciably above 200 C, and the covered portions of the paste will remain below the decomposition temperature. It is also possible to protect the joint area of the sensor by covering the external area with a protective layer of plastic (silicone, teflon, wax) to modify the heat transport in the area of the joint.

Indium solder may be used to solder leads directly to an adherent platinum film. The melting point of the indium solder is 150 C, and this temperature must be the limit of the calibration cycle. If the joint is buried under other materials, protection is provided against transient excursions to higher temperatures.

Lead/tin solders may be obtained that have melting points in the range of 225 C to 275 C. The previously stated cautions apply here, also. One problem

encountered with both tin/lead solders and indium solder is the difficulty in removing the solder if the gage body were to be reworked. The soldered area showed traces of oxidized solder after annealing, and it was difficult to attach a lead to this area reliably.

Silver solders are available with melting points ranging from 620 C to at least 845 C. Handy and Harman's Easy-Flo brazing alloy meets the temperature requirements of this task. Easy-Flo melts at 625 C and flows at 635 C.[10]

The preferred procedure for attaching leads to the gage with the Easy-Flo consists of the following steps. Grooves are cut into the cylinder surface of gage parallel to the axis of the cylinder. The groove width should be cut to barely clear the diameter of a platinum lead wire of 0.5 mm diameter. The groove will extend forward from the base for 1 cm. The groove is coated with the platinum film at the same time as the platinum film leads are developed for the sensor element. Platinum wires (0.5 mm) are tinned with the Easy-Flo alloy and bound into the grooves with nichrome wire. No flux is required. The gage is inserted into a furnace in such a manner that the substrate is heated more rapidly than the wires. An alternate procedure is to place the gage in a cold furnace and shield the leads by covering them with an insulator. The important point is to heat the substrate and the leads at the same rate or to heat the substrate at a faster rate. The liquid brazing alloy will flow towards the hottest point, and it is necessary to have the wire and the joint at nearly the same temperature.

After the gage has cooled, nickle lead wires may be spot welded to the short platinum lead wires. Copper or nichrome wires may also be used, but the copper tends to oxidize appreciably during calibration tests.

An important technique is used commercially to strengthen the attachment of the wire to the gage body. A glass frit is fused over the platinum lead and the substrate. This gives a durable connection that is independent of the electrical connection. I have not as yet obtained a good glazed film, but the technique is basically simple. It should be possible to combine the furnace brazing operation and the enameling process as one operation.

Spot welding of a thin foil of platinum to the platinum film may be possible, but I believe that simpler techniques are available.

IX. CALIBRATION OF FOUR WIRE RESISTANCE GAGES

Calibrations were based on platinum resistance thermometers with a specified deviation from calibration tables of ±0.1% on resistance and ±0.6 C/100 C. The units were 100 ohm platinum RTD elements from Omega Engineering, Inc. Appreciably better accuracy (0.1 C) is possible[11] with special platinum thermometers that have been calibrated for use as standards.

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[10] *"The Brazing Book," Handy and Harman, New York, 1977.*

[11] *"Manual on the Use of Thermocouples in Temperature Measurement," American Society for Testing and Materials, Philadelphia, Special Technical Publication 470A, 1974.*

The resistance of the gages was measured in the four wire configuration using a Hewlett-Packard Digital voltmeter, model 3455A. This system, when used in Auto Cal mode in "High Resolution," is capable of measuring the gage resistance to ±0.0046% or about ±.005 ohm.[12]

The linear coefficient of the reference gage at 100 C is 0.385 ohm/C.[7] A large aluminum block with drilled holes for the gages was used as a heat sink. The block was heated on a hot plate or in a furnace and then wrapped in foil backed fiberglass insulation.

Naturally slow cooling (100 C/hr) occurred over a period of hours as the calibration readings were taken. Leads to the gages were covered by fiberglass insulation for a distance of approximately seven centimeters. Manual recording of the resistance readings was performed. An automatic, computer controlled system capable of monitoring six to twelve gages was designed and fabricated by the personnel of the recording room. Conversion of the resistance values to temperature readings for the gages will be handled by the computer from stored tables.

Calibration curves of the standard linear relation of resistance change to temperature change are generated by linear regression.

X. CALIBRATION OF THE SUBSTRATE MATERIAL

G.T. Skinner's technique[5] of calibrating the backing material may be used to determine the parameter required in heat flux determination.

Basically, Skinner's technique consists of monitoring the temperature of a gage element as a heat flux pulse is applied. If identical heat flux pulses are applied to the gage in air and to the gage submerged in a liquid of known thermal properties, the properties of the substrate may be determined.

As in Skinner,[5] water may be used as the reference liquid if the thin film element is protected against the water. In the present case, no protective layer was used, so a nonreactive liquid must be found. Relevant thermal parameters are available for toluene.[13]

A variation of the technique allows the determination of the temperature dependence of the thermal parameter. The gage is pulse heated in a furnace over the range of temperatures required. The input to the gage is adjusted so that the heat flux will be the same at the various temperatures. This allows the parameter calculation based on the room temperature values.[5]

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[12] *"Operating and Service Manual, Model 3455A, Digital Voltmeter," Hewlett-Packard Company, Loveland, CO, 1976.*

[13] *J. Nagasaka and A. Nagashiwa, "Simultaneous Measurement of the Thermal Conductivity and the Thermal Diffusivity of Liquids by the Transient Hot-Wire Method," Rev. Sci. Instran., Vol. 55, No. 2, February 1981.*

The pulse heating is limited to a fraction of a degree, and the heating time is 10 ms to 100 ms.

XI. MATERIAL TESTS

A. Macor

Macor samples were dry polished with alumina compounds and chemically cleaned with Balzers substrate cleaners.

A critical step was found to be a heating cycle before the application of sputtered platinum film. Thermal cycling of the Macor to 800 C apparently removes moisture from the material.

The sample to be sputtered is sputter etched under an aluminum screen. The sample must be moved during the etch cycle to expose the entire surface.

The sputtering process deposited a layer of platinum over the entire body surface, including the lead wire holes/slots.

The sample was then subjected to a dry polishing operation to remove platinum in the areas required to define the sensor area and establish the lead areas.

The film annealing cycle was taken to somewhat higher temperatures than in the case of Pyrex based gages. 800 C (+) was utilized in an effort to stabilize the gage calibrations.

Various workers report that platinum gages on Pyrex are sensitive to moisture, and it is common practice to coat the gage area with silicon monoxide (SiO). Early work with the Macor gages included some effort at calibration to low temperatures (-50 C). In these tests, water vapor condensed on the gage body, and the gage calibration changed rapidly. Repeated annealing to high temperature and avoiding condensation appears to yield a stable calibration (Figure 1, the lowest trace).

The Hanovia Liquid Platinum may be sprayed onto Macor with an airbrush or may be applied by hand brushing.

Major problems were encountered with Macor. The books of mica in the Macor are 8 to 25 micrometers wide. Obtaining a good polish that is smooth and scratch-free is difficult due to the delamination of the mica and possibly due to included air bubbles. Some of the apparent water sensitivity of the platinum film may be due to the mica particles on the surface absorbing water and water that is a natural part of the mica structure. The bake-out cycle to remove the water was a critical step in achieving a relatively stable calibration.

B. Pyrex

The Pyrex was formed into the gage configuration by careful melting of a hemispherical end on the rod. Annealing of the Pyrex is required before the application of the platinum film.

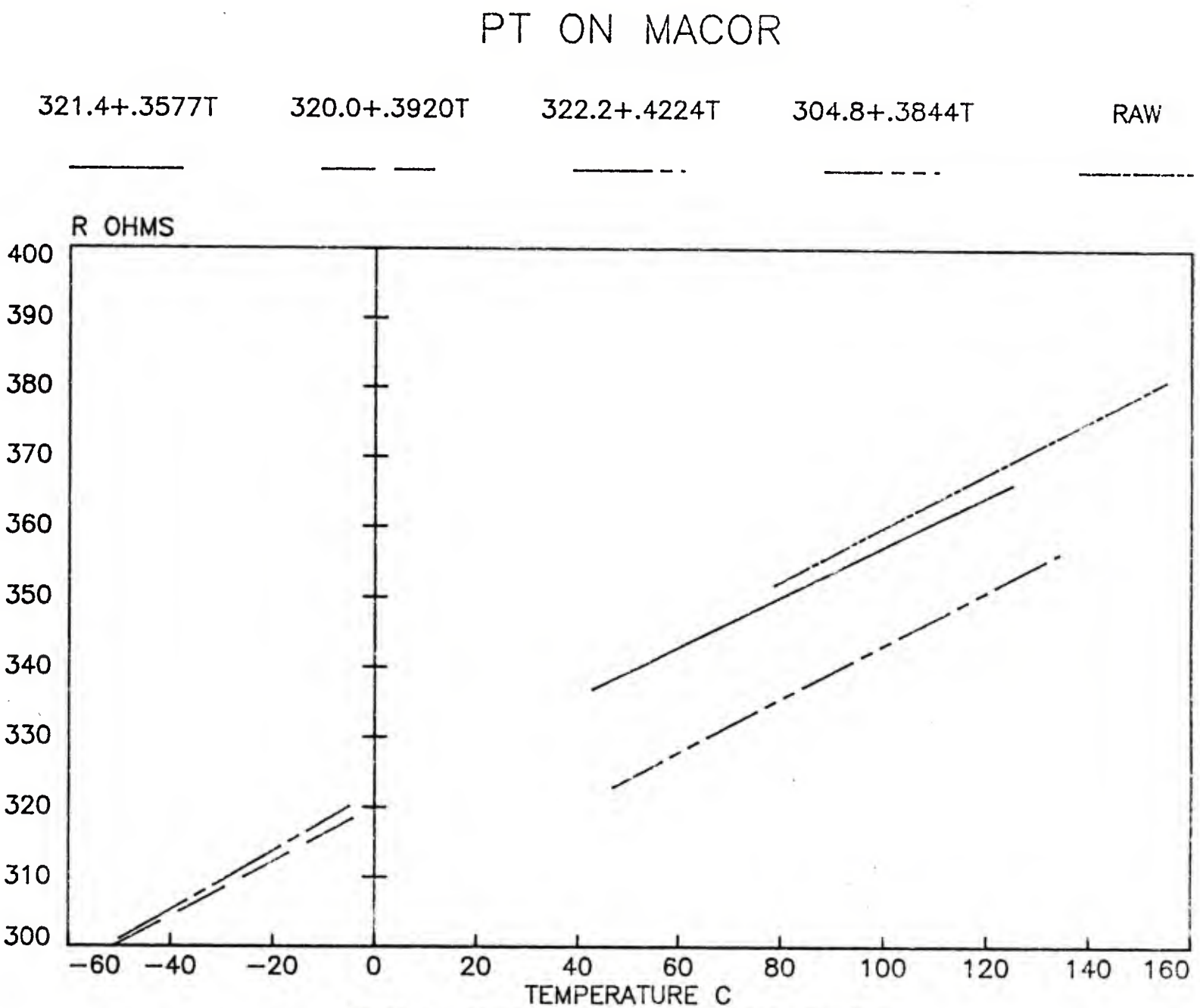

Figure 1. Sputtered Platinum On Macor, One Gage

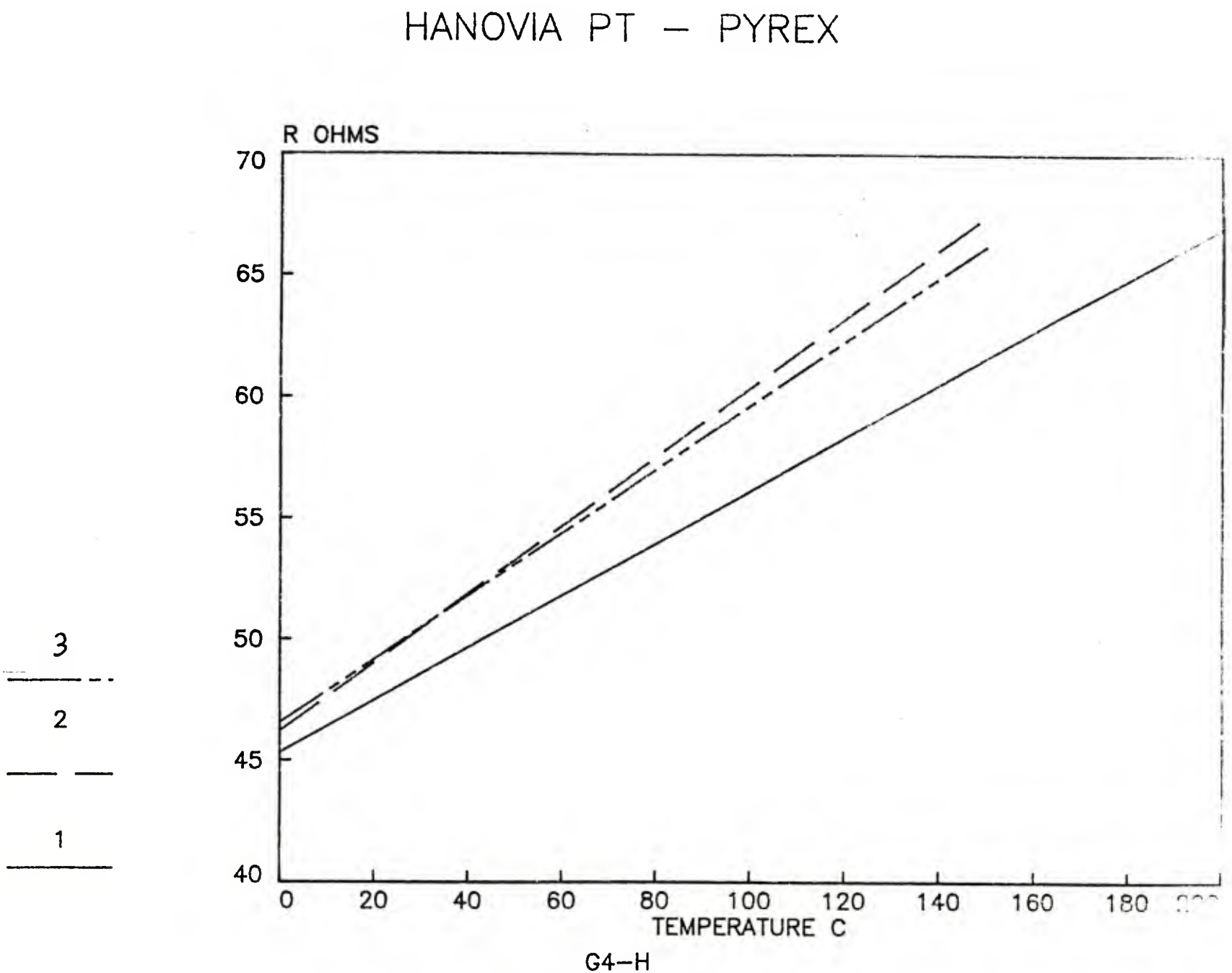

Figure 2. Platinum On Pyrex

Platinum films were coated onto Pyrex substrates by pyrolysis of Hanovia Liquid Platinum and by sputtering techniques. Both techniques produced gages that were acceptable for the convective ignition studies.

Table 2. Physical Properties, Macon

Temp	ρ g/cm^3	C_p cal/g C	k Conductivity	β^2 $\rho k C_p$
20 C	2.52	.181	3.08×10^{-3}	0.00140
100 C	2.51	.202	3.09×10^{-3}	0.00157
200 C	2.51	.222	3.10×10^{-3}	0.00173
300 C	2.50	.233	3.21×10^{-3}	0.001870
400 C	2.49	.237	3.23×10^{-3}	0.001906

Measured at 27C, our sample, ρ = 2.537 g/cm^3.
Coefficient of thermal expansion, 9.4 x $10^{-6}C^{-1}$.
Maximum (no load) use temperature: 1000 C.
Continuous use temperature: 800 C.
β :*Heat flux parameter cal^{2}cm^{-4}s^{-1}K^{-2}*

The films produced by the pyrolysis technique appeared to be more durably bonded to the Pyrex. Literature study indicates that the bonding could be improved by sputtering a precoat layer of another metal onto the substrate.[8,14] This metal (e.g., Al, Cr ...) chemically bonds to the substrate. With the available apparatus, this approach was not practical.

Figure 2 shows calibration curves for Hanovia platinum on Pyrex. All three traces are for the same gage but at various times in the life-cycle testing of the gage.

The following gage calibrations were obtained.

First calibration:

R = 45.308 ohm + 0.1086 T (C)

α = 0.002397 C^{-1}

Correlation: 0.99996

[14] *J. Lindmayer, "Arc Spraying Solderable Tabs to Glass," NASA Tech Briefs, Winter, 1980.*

Recalibration after use in conductive ignition tests, cleaned with trichloroethane, but not annealed again:

$R = 46.200 \text{ ohm} + 0.1418T \text{ (C) ohm}$

$\alpha = 0.002637 \text{ C}^{-1}$

Correlation: 0.99997

Values after another series of conductive ignition tests, no film annealing cycle applied:

$R = 46.549 \text{ ohm} + 0.1311T \text{ (C)}$

$\alpha = 0.002817 \text{ C}^{-1}$

Correlation: 0.99998

Table 3. Physical Properties, Pyrex

From Birk,[1] $\rho C_p k = 0.0013 \text{ cal}^2/\text{cm}^4 \text{ sec C}^2$

Skinner[5] lists the values of ρ, C_p, and k as:

$k = -0.035^2 + 0.00245 \log T \text{ (K) cal sec}^{-1} \text{ cm}^{-1} \text{ K}^{-1}$

$C_p = 0.174 + 0.00036 T \text{ (C) cal g}^{-1} \text{ K}^{-1}$

$\rho = 2.23 \text{ g cm}^{-3}$

$\beta^2(28 \text{ C}) = \rho C k = 0.001304 \text{ cal}^2 \text{ cm}^{-4} \text{ S}^{-1} \text{ C}^{-2}$

β is here stated to be accurate to ±5%. The range of temperature for the above data is not stated in the Skinner paper.[5]

Thermal expansion coefficient, 0-300 C: $3.5 \times 10^{-6} \text{ C}^{-1}$

Upper working temperature (annealed): 490 C

Annealing point: 565 C

Softening point: 820 C

C. Boron Nitride

A boron nitride sample was prepared for sputtering by polishing with dry toilet paper. The sample was spun in a drill press as the paper was wiped over the surface. After a polish was obtained and grooves for the lead wires cut in the base of the sample, the sample was sputter etched under an aluminum screen with movement of the sample during the etch cycle (15 minutes, 10 Pa argon, 37 mA).

The sample was then sputtered with platinum. The sample was placed with the hemispherical end near the sputter source to reduce the concentration on the tip while depositing a thicker layer along the sides for leads. The sample was then inverted and the sensor end wrapped with aluminum foil. Platinum was applied in this orientation to give thicker leads and coat the grooves. The sample was placed in a furnace and "annealed" to 700 C.

The resulting film was well attached to the sample.

Boron nitride, hot pressed 96%. (Anisotropic properties are determined by the direction of pressing.[15])

Specific Heat (cal/gC)

182C: .278
266C: .312
454C: .307
682C: .373

Thermal Conductivity (cal cm/cm^2 s C)

	Parallel	Perpendicular
315 C	0.04375	0.0875
437 C	0.0424	0.0847
557 C	0.0410	0.0820
710 C	0.0386	0.0772
978 C	0.0372	0.0744

Thermal expansion coefficient:

0-700C 8.06×10^{-6} C^{-1} parallel to press
0.882×10^{-6} C^{-1} perpendicular to press

24C-350C 10.15×10^{-6} C^{-1} parallel
0.594×10^{-6} C^{-1} perpendicular

Ratio = 17.1

Density 2.10 g/cm^3 TMD 2.25 g/cm^3

Long-term exposure to high humidity (81% RH) gives a weight gain of about 1%. A possible attack of Pt and Ni on BN may be observed.

D. Aluminum Silicate

A number of materials are available that are machinable as received and may be fired to produce a hard ceramic material. Steatite is a natural mineral that is a hydrous magnesium silicate. It is commonly used to make

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[15] *"Boron Nitride, Mechanical and Refractory Properties," Technical Data File 1B164 Carborundum, Latrobe, PA.*

high-voltage insulators for experimental purposes. A hydrous aluminum silicate is available commercially (American Law Corporation, Chattanooga, TN). This material (Lava, Grade A) may be cut with common tools and is hardenable by firing at 1000 C. It is appreciably easier to work with than the Macor ceramic and lacks the mica inclusions of Macor. For fabrication purposes, it is preferable to Macor or Pyrex.

Cutting and polishing in the unfired state are relatively easy with standard shop equipment. The Lava is hardened by firing to 1000 C to 1100 C. After cooling, a final polishing operation was performed with alumina powder on paper. A good polish was readily achieved.

A chemical cleaning cycle in hydrochloric acid was used as in the Pyrex trials. The samples were then dried by heating.

A platinum film was sputtered on as described for previous materials. Firing the resulting gage to about 700 C resulted in a platinum film that was adherent under handling and tape peel tests.

Aluminum Silicate (Iava, Grade A)

Thermal conductivity 0.003 cal cm/cm^2 s C 0-300 C

Density: 2.35 g/cm^3 (Fired)

Specific heats: NA

Safe limit temperature: 1100 C

Softening temperature: 1600 C

Unfired hardness: 2 (Mhos scale)

Fired hardness: 6 (Mhos scale)

E. Vespel

A sample of Vespel was tested for adhesion of a thin gold film. The Vespel sample was sanded flat and polished with tripoli and alumina agents applied to rag paper sheets. Balzers substrate cleaning compounds were used to chemically clean the surface. The sample was masked with ester tape to define sensor and lead areas. Sputter etching under an aluminum screen was used as the final cleaning operation. Gold was sputtered on the sample (3 min., 20 MA, 50 nm). The mask was stripped from the sample. The sample was annealed at 300 to 350 C to stabilize the film.

A bright, adherent film resulted. The resistance of the film strip was adequate for gage use. No film or substrate calibrations were attempted at this time.

Vespel Polyimide from Dupont

Density: 1.43 g/cm^3

Thermal conductivity: 3.3 to 3.7 x 10^{-3} J/cm^2 s C/cm
.79 to .89 x 10^{-3} cal/cm^2 s C/cm

Specific heat: 0.27 cal/gC
1.13 J/g C

Compressive strength: 275 MPa

XII. SUMMARY DISCUSSION

Adherent metal films were obtained on Macor, Pyrex, Lava, and Vespel by sputtering or pyrolytic techniques.

Both processes may be seen to consist of polishing, chemical cleaning, drying, applying the film, and annealing the film. The pyrolytic technique produces good films on glasses as long as the temperature of the firing sequence is well controlled and the layer of Hanovia liquid is not too thick. The sputtering process yields thicker films more easily and produces films with initially (at least) good adherence if a proper sputter etch cycle has been used as the last cleaning step before film deposition.

The pyrolytic technique with glasses is fairly easy to master and will provide acceptable gages.

The Macor should probably be avoided for heat flux gage use unless much more complicated shapes are required. The difficulty is met in polishing the material, the water sensitivity of the calibration, and the tendency of the mica particles to flake from the surface. The flaking of the mica under the platinum film allows the initiation of peeling of the platinum that extends beyond the area of the mica. Only one reference[16] was found in which Macor had been used in heat flux gage fabrication. This work was limited to 100°F calibrations and does not discuss details of reproducibility or constancy of the gage factors. From the manufacturer's data curves, it appears that a crystal transition may be occurring near 300 C. Dynamic calibrations should be made through this temperature before static calibrations are accepted for heat flux gage use.

The ease with which Pyrex may be fabricated in the shape required for the convective ignition work and the extent of the experience in using this material for heat flux gages dictates that it be the first choice for gage

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[16] *H.A. Heperekan, "Experimental and Theoretical Study of Heat Transfer with Combustion," LBL-10868, LBL, California University, Berkeley, CA, May 1980.*

substrates. Higher melting glasses such as Vycor or fused quartz should be as easy to use and would extend the temperature range somewhat.

The great anisotropy of the boron nitride material rules against its use as a substrate for this study. The computer program[17] for obtaining the heat flux assumes isotropic properties of the substrate material near the gage.

Two candidate materials should be evaluated more carefully.

The aluminum silicate, Lava, Grade A, fabricates and polishes easily. In its hardened state, it appears to accept a platinum film readily.

The Vespel plastic appears to accept a gold film readily. Its thermal properties are closer to those of a propellant, and its use temperature should be near the "ignition" surface temperatures of propellants under transient conditions. More work is required to judge the stabilizing of the gold film calibration at various use temperatures.

XIII. CONCLUSIONS

For the convective ignition tests, the basic substrate material for heat flux gages should be selected as Pyrex, Vycor, or fused quartz, with a preference for Pyrex due to its use history.

An aluminum silicate, Lava, Grade A, and a plastic, Vespel, should be tested for further application.

The gage should be fabricated in the four wire configuration in order to minimize lead heating errors.

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[17] *W.J. Cook and E.J. Feldman, "Reduction of Data from Thin Film Heat Transfer Gages...," AIAA Journal, Vol. 4, No. 3, p. 561, March 1966.*

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LIST OF SYMBOLS

C_p - specific heat cal/gC

k - thermal conductivity cal/cm^2 C

R - resistance, ohms

T - temperature, C or K

TCR or α - temperature coefficient of resistance C^{-1}

t,w,l - thichness, width, length

β - heat flux constant $\equiv (\rho k C_p)^{1/2}$

ρ = density (g cm^{-3}) or resistance (ohms)

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